Effect of Zoe Temporary Restoration on Resin-Dentin Bond Strength Using Different Adhesive Strategies

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ABSTRACT

Statement of the Problem: Eugenol is a radical scavenger that inhibits the polymerization of resin materials. Little is known about the effect of a eugenol-containing temporary restorative material on the resin-dentin bond strength of adhesive systems that partially dissolves and modifies the smear layer.

Purpose of the Study: The aim of this study was to evaluate the effect of eugenol-containing temporary restoration (zinc oxide eugenol [ZOE]) on the resin-dentin microshear bond strength of etch-and-rinse and self-etch adhesives.

Material and Methods: The roots of 18 human molars were removed and the crowns of the teeth were transversally sectioned into two halves. The dentin surfaces were embedded in acrylic resin. Half of the samples were stored while the remaining specimens were restored with eugenol-containing temporary restoration. After 24 hours, the ZOE restoration was mechanically removed and dentin surfaces were ultrasonically cleaned. Dentin surfaces were treated with one of the following adhesives: Single Bond, Clearfil SE, and iBond. Six cylinders of Z250 (0.5 mm high and 0.75 mm in diameter) were applied to each bonded dentin surface using a tygon tube. After storage for 24 hours, the specimens were subjected to micro-shear testing. The data was subjected to a two-way analysis of variance and Tukey's test ($\alpha = 0.05$).

Results: Similar bond strength values were obtained for Single Bond ($p = 0.48$) either in the control or in the ZOE-treated group. For both self-etch systems, the bond strength in the ZOE-treated group was statistically lower than the control group ($p < 0.01$).

Conclusions: Although the prior use of eugenol-containing temporary restoration (IRM) affects the resin-dentin bond strength of the etch-and-rinse Single Bond, a more pronounced reduction on bond strength was observed for the two self-etch systems evaluated (iBond, Clearfil SE Bond).

CLINICAL SIGNIFICANCE

Eugenol-containing provisional restorations (IRM) should not be used prior to the placement of resin restorations bonded with the two-step etch-and-rinse Single Bond and the self-etch adhesives systems iBond and Clearfil SE Bond.

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INTRODUCTION

The placement of tooth-colored restorations has increased substantially over the last several years because of the improvement in formulations and increased aesthetic demands by patients and clinicians. The degree of complexity of composite resin restoration placement is higher than that of amalgam and, in a few cases, the lack of clinical time prevents the placement of composite restoration in just one clinical appointment. Therefore, the application of a temporary material is indispensable for sealing the cavity until the next appointment.

Among the temporary restorative materials, zinc oxide eugenol (ZOE) is presumably one of the most commonly used temporary material for endodontics and restorative dentistry because of its sedative effect on sensitive teeth, low cost, ease of removal, and excellent seal against leakage.[1] When zinc oxide is mixed with eugenol, in the presence of a small amount of water, a chelation reaction takes place and results in a set mass of unreacted zinc oxide particles in a matrix of zinc eugenolate. Unfortunately, this reaction is reversible (ie, when the set cement contacts water, the eugenolate at the surface hydrolyzes to liberate eugenol).[2]

Thus, eugenol released from ZOE mixtures can penetrate dentin[3, 4] and interact with resin-based restorative materials. As other phenolic compounds, eugenol is a radical scavenger that inhibits the polymerization of resin materials.[5] The hydroxyl group of the eugenol molecule tends to protonize the free radicals formed during the polymerization of resin-based materials, thereby blocking their reactivity[6] and reducing the degree of conversion of these materials.[7]

Contradictory findings exist regarding whether or not the prior use of eugenol-containing temporary restorations affects the bond strength of composites to dentin. While some studies reported that eugenol-containing temporary restorations should be avoided in such cases,[8–11] other studies have not observed any detrimental effect on bond strengths when eugenol-based materials are used.[12–16]

It is worth emphasizing that most of the aforementioned studies have employed earlier generations of bonding systems and etch-and-rinse adhesive systems. Few studies have attempted to evaluate the effect of eugenol-based materials on the bond strength of newer self-etch adhesive formulations.[12] As the smear layer is not removed with acids prior the application of self-etch adhesives, it is likely that more remnants of eugenol molecules and residues of eugenol-containing temporary restoration will be incorporated into the hybridized complex, which may affect the bonding performance of these new systems.

Therefore, the aim of this study was to evaluate the influence of a eugenol-containing temporary restoration on the bonding of an etch-and-rinse adhesive system (Single Bond, 3M ESPE, St. Paul, MN, USA) and two self-etch adhesive systems that partially dissolve and modify the smear layer (Clearfil SE Bond [Kuraray Medical Inc., Tokyo, Japan] and iBond [Heraeus Kulzer, Hanau, Germany]). The null hypothesis to be tested is that the prior use of eugenol-containing temporary restoration will not affect the bond strength of the three adhesives evaluated.

MATERIALS AND METHODS

Eighteen extracted, caries-free human third molars were used. The teeth were collected after the patient's informed consent. The University of São Paulo Institutional Review Board approved this study. Teeth were disinfected in 0.5% chloramine, stored in distilled water at 4°C, and used within 6 months after extraction. The roots of all teeth were removed by sectioning with a diamond saw at slow speed (Isomet, Buehler, Lake Bluff, IL, USA) under water irrigation (Figure 1). The crowns of the teeth were transversally sectioned into two halves (Isomet) and the dentin surfaces were examined in a stereomicroscope at 40× (HMV-2,

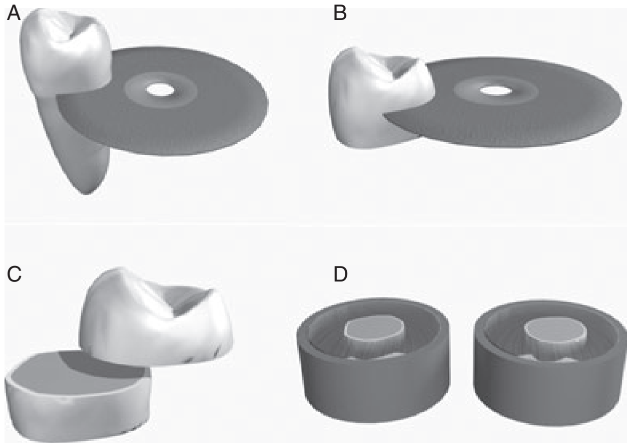

Figure 1. Schematic drawing showing the preparation of the specimens for bonding. (A) First, the roots were removed and then (B) a transversal sectioning was performed in order to obtain two tooth crown halves. (C, D) Both sections were embedded in acrylic resin in a polyvinyl chloride tube.

Shimadzu, Tokyo, Japan) to ensure that they were free of enamel remnants (Figure 1). Then, both tooth halves were embedded in polyvinyl chloride (PVC) tube using acrylic resin (Jet, Artigos Odontológicos Clássico, SP, Brazil) (Figure 1). The enamel-free, exposed dentin surfaces were further polished on wet #600-grit silicon-carbide paper for 60 seconds to standardize the smear layer. The specimens were then ultrasonically cleaned in distilled water for 5 minutes prior to the bonding procedure to remove any remaining silicon carbide dust particles.

One tooth half (control) received no temporary restoration. The other tooth half was restored with ZOE (IRM, Dentsply, Petrópolis, Brazil). The ZOE was mixed at the manufacturer's recommended powder: liquid (P:L) ratio (six parts powder:one part liquid); the weight was measured on an analytical balance (Ohaus, Shangay, China). The cement was placed in the dentin surface and pressed against the surface by means of a glass slide. Following a 1-hour set, the specimens were stored in distilled water at 37°C for 24 hours. After the storage period, the ZOE-containing temporary restorations were mechanically removed with a scaler until the dentin surfaces were visually macroscopically free of material. The specimens were then cleaned with a pumice-water slurry (Pasom Materiais Odontológicos LTDA, SP, Brazil) in a slow-speed handpiece for 60 seconds and rinsed off with air-water stream (60 seconds), before adhesive application.

Three bonding systems were evaluated: Clearfil SE Bond, which is a two-step self-etch system; iBond, a one-step self-etch system; and Single Bond, a two-step etch-and-rinse adhesive system (Table 1). Six pairs of tooth halves were assigned for each adhesive system. The adhesive systems were applied on dentin surfaces according to the manufacturer's directions (Table 1). After applying the adhesive to the dentin, six vinyl Tygon tubes (TYG-030, Small Parts Inc., Miami Lakes, FL, USA) 0.75 mm in diameter and 0.5 mm high were placed on the dentin surface all at once and the adhesive was light-cured (10 seconds) thereby fixing the tubes to the dentin surface (Figure 2). Resin composite (Z250, shade A2, 3M ESPE) was placed in the tubes and light-cured for 40 seconds (Optilux 500, Demetron, Danbury, CT, USA) with a power density of 600 mW/cm^2 (Figure 2). The power density of the curing device was regularly checked with a curing radiometer (Demetron, Orange, CA, USA). The specimens were stored in water at 37°C for 24 hours. The vinyl tubes were removed with a blade and then checked with a light stereomicroscope at 10× magnification to discard any specimens with air bubbles or gaps evident at the interface (Figure 2). The flash of composite resin extending beyond the base of composite resin was removed with a blade.

TABLE 1. COMPOSITION, APPLICATION MODE, AND BATCH NUMBER OF THE MATERIALS AND ADHESIVE SYSTEMS EMPLOYED.

Materials/Systems	Composition	Application mode	Batch number
Single Bond (3M ESPE, St. Paul, MN, USA)	1. 35% phosphoric acid 2. Adhesive—Bis-GMA, HEMA, dimethacrylates, polyalknoic acid copolymer, initiators, water, and ethanol	1—Acid etching (15 seconds), rinsing (15 seconds), and air-drying (10 seconds) leaving dentin moist 3—Application of one coat of the adhesive (10 seconds with slight agitation) 4—Air-dry (10 seconds at 20 cm) 5—Application of one coat of the adhesive (10 seconds with slight agitation) 6—Air-dry (10 seconds at 20 cm) 7—Light-activation (10 seconds—600 mW/cm^2)	4JR
Clearfil SE Bond (Kuraray Medical Inc., Tokyo, Japan)	1. Primer—water, MDP, HEMA, camphoroquinone, hydrophilic dimethacrylate 2. Adhesive—MDP, Bis-GMA, HEMA, camphoroquinone, hydrophobic dimethacrylate, N,N-diethanol p-toluidine bond, silanated colloidal silica	1—Application of two coats of the primer with slight agitation (20 seconds) 2—Air-dry (10 seconds at 20 cm) 3—Application of one coat of the adhesive (15 seconds) 4—Air-dry (10 seconds at 20 cm) 5—Light-activation (10 seconds—600 mW/cm^2)	00447A 00593B
iBond (Heraeus Kulzer, Hanau, Germany)	1. 4-META, UDMA, acetone, water, glutaraldehyde, camphorquinone	1—Application of three consecutive coats of the adhesive, brushing for 10 seconds each 4—Air-dry (10 seconds at 20 cm) 5—Light-activation (20 seconds—600 mW/cm^2)	010066
Filtek Z-250 (3M ESPE)	1. Filler type—zirconia, silica 2. Resin—Bis-GMA, UDMA, Bis-EMA	1—Light-activation (40 seconds—600 mW/cm^2)	4BC
IRM (Dentsply, Petrópolis, Brazil)	1. Powder—zinc oxide, PMMA powder 2. Liquid—eugenol, acetic acid	1—Mixed at manufacturer's instruction for 60 seconds	206495

4-META = 4-methacryloloxyethyl trimellitate anhydride;
bis-EMA = ethoxylated bisphenol A dimethacrylate;
bis-GMA = bisphenol A diglycidyl methacrylate;
HEMA = hydroxy ethyl methacrylate;
MDP = 10-methacryloyloxydecyl dihydrogen phosphate;
PMMA = polymethyl methacrylate;
UDMA = urethane dimethacrylate.

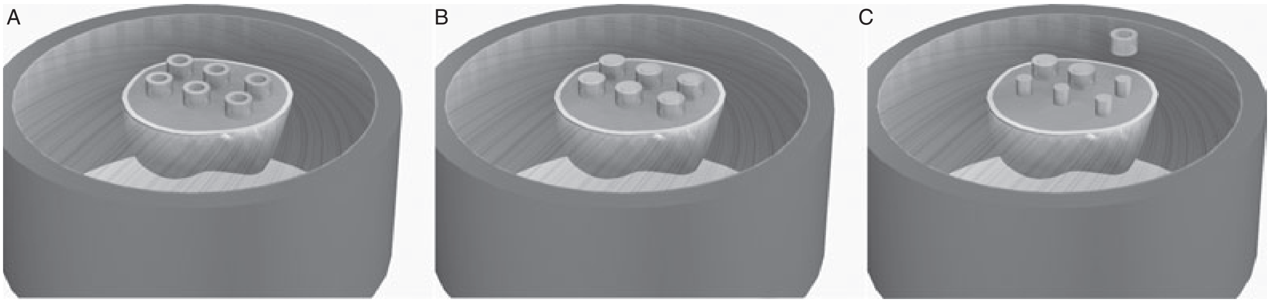

Figure 2. Schematic drawing showing specimen preparation for microshear testing. After application of the adhesive systems, the vinyl Tygon tubes were placed on the dentin surfaces (A) and the tubes were filled with a composite resin (B). (C) shows the aspect of the dentin after partial removal of vinyl tubes.

A universal testing machine (Instron Testing Machine-Model 5565, Instron, Canton, MA, USA) was used for the microshear bond test. Each PVC tube containing the bonded specimens was attached to the testing device (Figure 3), which, in turn, was placed in the universal testing machine. A thin wire (0.2-mm diameter, Morelli Ortodontia, São Paulo, Brazil) was looped around the composite resin cylinder, around half its circumference, and gently held flush against the dentin at the resin-dentin interface (Figure 3). A shear force was applied to each specimen at a crosshead speed of 0.5 mm/min until failure occurred. The force required to failure was then divided by the bonded area of the vinyl tube and the bond strength values expressed in MPa. The resin-dentin interface, the wire loop, and the center of the load cell were aligned as straight as possible to ensure the correct application of the shear force.

Following the microshear bond test, the fractured specimens were examined in a stereomicroscope (HMV-2) at 40× and the failure modes were classified as: mode 1, adhesive failure at the adhesive and dentin interface; mode 2, cohesive failure within the composite; and mode 3, cohesive within dentin, if the fracture occurred exclusively in dentin.

Six tooth halves were employed and in each tooth half, six resin cylinders were constructed, allowing bond strength measurement in 36 different sites. The bond strength values obtained in each tooth half were averaged for statistical purposes. The data (MPa) were subjected to a two-way analysis of variance (ANOVA) and Tukey's test at a significance level of 0.05.

RESULTS

The mean microshear bond strength values and the respective standard deviations are shown in Table 2. The percentages of fracture modes in all experimental groups are shown in Table 3.

The results from two-way ANOVA revealed that the interaction eugenol pretreatment versus adhesive was not statistically significant ($p > 0.05$), but the main factors eugenol pretreatment ($p = 0.003$) and adhesive ($p = 0.001$) affected significantly the mean bond strength. Significant reductions on bond strengths were observed when dentin was pretreated with eugenol-containing temporary restoration ($p = 0.003$). However, the reduction observed for the etch-and-rinse Single Bond (9.6%) was less pronounced than that observed for Clearfil SE Bond (22.3%) and iBond (22.1%).

Comparing the different adhesives' performance, the two-step etch-and-rinse Single Bond and the two-step self-etch Clearfil SE Bond showed similar performance ($p > 0.05$). The lowest resin-dentin bond strength values were observed by the one-step self-etch iBond ($p < 0.01$).

DISCUSSION

The contemporary resin-based adhesives can be classified based on the underlying adhesion strategy as etch-and-rinse and self-etch adhesives.[17] Two-step etch-and-rinse adhesives, as Single Bond, require the pretreatment of dentin with an

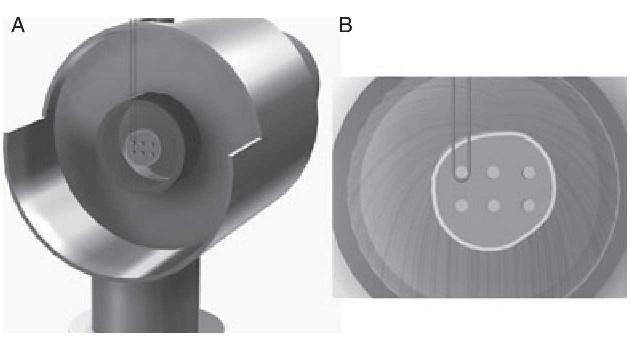

Figure 3. Schematic drawing showing the specimens immediately before testing. A, The polyvinyl chloride tube was attached to the testing device. B, Thin wire looped around the composite resin cylinder for the microshear testing.

TABLE 2. MEAN MICROSHEAR BOND STRENGTH VALUES (MPA) AND THE RESPECTIVE STANDARD DEVIATIONS (±SD) OF ADHESIVE SYSTEMS TO DENTIN WITH OR WITHOUT PRETREATMENT WITH ZINC OXIDE EUGENOL (ZOE).

Adhesive systems	Control	ZOE-treated
Single Bond	31.3 ± 2.7a	28.3 ± 3.8b
Clearfil SE Bond	30.5 ± 2.0a	23.7 ± 1.7c,d
iBond	25.3 ± 5.7c	19.7 ± 8.5d,e

Different letters indicate statistically different means.

TABLE 3. PERCENTAGE OF FAILURE MODES (%).

Adhesive systems	Control			ZOE-treated		
	Mode 1	Mode 2	Mode 3	Mode 1	Mode 2	Mode 3
Single Bond	87.5	—	12.5	100	—	—
Clearfil SE Bond	92	—	8	100	—	—
iBond	100	—	—	100	—	—

Mode 1 = adhesive; Mode 2 = cohesive in resin; Mode 3 = cohesive in dentin.

acid. This acid, usually 30 to 40% phosphoric acid, superficially demineralizes dentin and thereby exposes a 3 to 5 µm collagen scaffold. Then, a solvent-rich, hydrophilic adhesive is applied on the demineralized dentin, and it diffuses into the collagen fibril's nanopores and forms the hybrid layer after in situ polymerization.[18]

In the self-etch approach, the infiltration of resin into dentin occurs simultaneously with the etching process. In these systems, the adhesive resin should penetrate beyond the smear layer and etch the intact underlying dentin to form a true hybrid layer.[17] Therefore, the smear layer is not removed beforehand but is incorporated into the hybrid layer complex. The present investigation demonstrated that the pretreatment with eugenol-containing provisional material caused reductions on resin-dentin bond strength values for both adhesive strategies, which led us to reject the null hypothesis of this study. However, this reduction on bond strength values after pretreatment with eugenol-containing material was much more pronounced for the two self-etch adhesives. This finding partially agrees with a previous investigation that compared these two bonding strategies after pretreatment with a eugenol-containing material.[12]

Some studies have reported that a thin layer (0.9–3.0 µm) of smear layer covers the dentin surface upon flat grinding.[19,20] Therefore, it is fair to suppose that when eugenol-containing temporary restoration was placed over the smear layer and left for 24 hours, eugenol probably leached into and through the smear layer to the dentin tubules, contaminating the dentin surface.[4] Hume[3] has found the concentration of eugenol in the aqueous phase to be in the order of 10^{-2} M just beneath the ZOE cement and 10^{-4} adjacent to the pulp.[21] This means that the concentration of eugenol is higher at the dentin surface near the ZOE cement decreasing toward the pulp. Contrary to the etch-and-rinse adhesive, the self-etch systems are applied directly over the contaminated smear layer dentin, which can explain why the reduction on bond strength was more pronounced for the self-etch bonding approach after pretreatment with eugenol-containing temporary restoration.

However, it is worth mentioning that other studies have not reached similar findings.[22–24] No significant reductions on resin-dentin bond strengths were observed after ZOE cement pretreatment. Unfortunately, two out of these three studies are abstracts,[22,23] which makes the analysis of the experimental design and the inherent variables from the studies difficult. The third article[24] employed conventional shear testing for measuring the resin-dentin bond strength of self-etch systems with and without previous contact with ZOE-containing restorative material. This test employs a larger area for bonding and therefore it is more sensitive to intrinsic defects in the bonded interface.[25] As a consequence, when the bond strength exceeds a threshold value (usually within 17–22 MPa), there is a trend toward cohesive rather than adhesive failure, as the truly interfacial strength is not being actually measured. The report on failure modes is, therefore, essential for the evaluation of the results from conventional tests and this report was not found in the study of Peutzfeldt and Assmussen.[24] Apart from that, the range of bond strength values that can be obtained when the failure occurs in the interface is lower for conventional tests than that observed for microshear or microtensile testing. This reduces the sensitivity of the method to detect small differences in bond strength means among experimental groups.

As can be seen in Table 3, a low number of cohesive failures were observed in the present investigation. This increases the internal validity of the data regarding the measurement of the true interfacial strength. The comparison of the failure modes of the Single Bond and Clearfil SE Bond with and without previous contact with eugenol-containing temporary material shows that adhesive failures were only observed in the eugenol-treated groups, which suggests that the interfaces from the latter groups were more fragile than the former. However, this data should be further evaluated because the present study did not attempt to provide a comprehensive evaluation of the fractured surfaces. They were only evaluated at 40× magnification by means of a stereomicroscope.

Some authors reported that the reduction on resin-dentin bond strength values after pretreatment with eugenol-containing materials is caused by the cement itself rather than eugenol, as remnants of temporary materials are not completely removed before adhesive application.[26–28] It was already demonstrated that the mechanical removal of provisional restorations with a dental probe,[28] the cleaning of the surface with a pumice slurry,[29] and also the etching with 37% phosphoric acid did not completely remove all the temporary restoration remnants from dentin.[28]

However, the phosphoric acid pretreatment eliminates the contaminated smear layer and results in the demineralization of dentin to a depth of 9 to 10 µm.[30] This depth of demineralization and the water rinsing after etching likely reduces the amount of free eugenol and temporary restoration remnants on the dentin surface.

Probably, the type of eugenol-containing temporary cement used may be the key to the controversy in the literature. There are four types of ZOE cements.[31] The great majority of the studies that evaluated the effects of eugenol-containing cements on resin-dentin bond strength employed Type I cements, intended for temporary luting procedures. This type of ZOE cement has a lower P:L ratio than Type III cement, indicated for temporary restorations and thermal insulating bases. Consequently, it is likely that Type I eugenol cement releases more eugenol, which can be responsible for the lower resin-dentin bond strengths shown by some investigators.[13,27,32,33] Few studies[14] have attempted to evaluate Type III eugenol-based cements as the present one.

In fact, it was already demonstrated that a P:L ratio lower than that recommended by the manufacturers can reduce the resin-dentin bond strength of etch-and-rinse adhesive systems. No significant reductions on resin-dentin bond strength values and microleakage were observed for a three-step etch-and-rinse system when a eugenol-containing temporary restoration was mixed in the recommended ratio (10 g:1 g) and used beforehand.[34,35] Opposite findings were observed, however, when the same adhesive was applied after pretreatment with a temporary material mixed at a 10 : 2 ratio. Although an excess of eugenol is usually within the eugenolate matrix, eugenol can be released from the eugenolate as soon as it comes into contact with free water. The wetter the ZOE mixtures (low P : L ratio), the higher the amount of free eugenol released into dentin.[4] However, the aforementioned authors have not evaluated self-etch adhesives. The results of the present investigation suggest that even recommended P : L ratios can impair bonding as long as self-etch adhesives are employed.

Although it was not the primary aim of the present investigation to compare the bond strength values of the different adhesive systems, the one-step self-etch system showed an inferior performance when compared with the two-step etch-and-rinse and self-etch systems. This finding was, in fact, confirmed by a recent review of the literature.[36] Statistical analyses of the pooled dentin microtensile bond strength data of a large group of commercial and experimental adhesives to dentin were performed.[36] According to this study, the three-step etch-and-rinse adhesives bonded significantly more strongly to dentin than did two-step etch-and-rinse and two-step self-etch adhesives. The two latter systems did not perform significantly different from each other. However, the significantly least favorable microtensile bond strength results were recorded for one-step self-etch adhesives, as shown by the present investigation. Therefore, these systems should be avoided by clinicians in a daily clinical practice.

CONCLUSIONS

Based on the results of this in vitro experiment, one can conclude that the pretreatment of dentin with a eugenol-containing temporary restoration affects the resin-dentin bond strength of the three adhesive systems evaluated, mainly for those based on the self-etch approach.

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